Reactions of Transition Metal Organyl Complexes with Olefins, 19[◇]

Photochemical Reactions of a Binuclear Naphthalene-Manganese Complex with 2-Butyne

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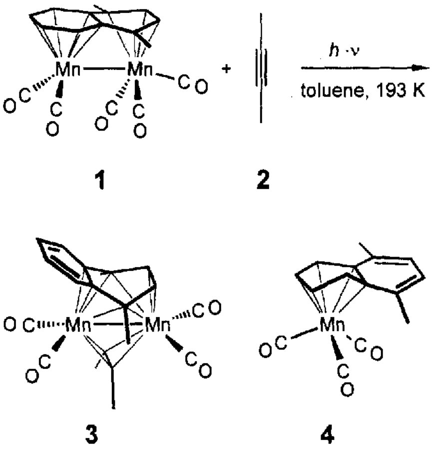
Pentacarbonyl(μ-$\eta^{4:6}$-1,4-dimethylnaphthalene)dimanganese-(*Mn*–*Mn*) (**1**), upon UV irradiation at 193 K in the presence of 2-butyne (**2**), yields tetracarbonyl(μ-$\eta^{2:2}$-2-butyne)(μ-1,2,3,4-η:1,4,4a,8a-η-1,4-dimethylnaphthalene)dimanganese (**3**) and tricarbonyl(4a,6,7,8,8a-η-5-hydro-1,4-dimethylnaphthalene)-manganese (**4**). The tetracarbonyl complex **3** rearranges in solution at room temperature to give tetracarbonyl(μ-$\eta^{2:2}$-2-butyne)(μ-4a,5,8,8a-η:5,6,7,8-η-1,4-dimethylnaphthalene)-dimanganese (**5**). The complexes **3** and **5** were characterised by IR and NMR spectroscopy, and by mass spectrometry, and complex **4** by IR and NMR spectroscopy. The molecular structures of **3** and **4** were determined by single-crystal X-ray diffraction analyses.

Arene complexes exist with almost every transition metal and in a variety of coordination modes[2][3][4]. The category of bimetallic complexes bridged by an aromatic ring system can be divided into *syn*-μ-$\eta^{m:n}$-arene complexes[5][6][7] and *anti*-μ-$\eta^{m:n}$-arene complexes[5][8][9][10]. The first μ-$\eta^{4:6}$-naphthalene-bridged *syn*-facial bimetallic complexes were isolated by Sweigart et al.[11]. The photochemical behavior of one of these complexes towards 2-butyne was the object of our studies.

Pentacarbonyl(μ-$\eta^{4:6}$-1,4-dimethylnaphthalene)dimanganese (**1**)[11], upon UV irradiation at 193 K in the presence of 2-butyne (**2**), yields tetracarbonyl(μ-$\eta^{2:2}$-2-butyne)-(μ-1,2,3,4-η:1,4,4a,8a-η-1,4-dimethylnaphthalene)dimanganese (**3**) and tricarbonyl(4a,6,7,8,8a-η-5-hydro-1,4-dimethylnaphthalene)manganese (**4**) (Scheme 1).

Scheme 1



In the formation of **3**, the initial loss of a CO ligand from the $Mn(CO)_3$ unit must be considered. After coordination of one alkyne molecule to the unsaturated complex has taken place, the formation of the dimanganatetrahedrane core is associated with a shift of the 1,4-dimethylnaphthalene ligand and a change in its coordination mode from μ-$\eta^{6:4}$ of both rings to μ-$\eta^{4:4}$ of only ring 1 to the two metal centres. The formation of **4** signifies the cleavage of the $[Mn(CO)_2]$ fragment from **1** and the addition of one hydrogen, possibly from the solvent.

Complex **3** crystallizes in the orthorhombic space group *Pbca*. Figure 1 shows an ORTEP projection[12] of the molecular structure of **3**.

The coordinated ring 1 of the 1,4-dimethylnaphthalene ligand and the two metal centres can be interpreted as being a *nido* cluster with eight vertices and 56 skeletal electrons. This species can be derived from a *closo* cluster in which an additional atom is located above the six-membered ring. This hypothetical *closo* cluster is closely related to the three-fold face-capped trigonal prism.

The carbon atoms C1, C2, C3 and C4, coordinated to Mn1, form a plane which has a distance of 183.5 pm to Mn1. Mn2 is at a distance of 189.3 pm from the plane formed by C1, C8a, C4a and C4. The 1,4-dimethylnaphthalene ligand is bent along the C1–C4 axis with a dihedral angle of 137.1°, whereas ring 2, which is only partially coordinated with C4a and C8a, remains planar. The Mn1–Mn2 distance of 241.3 pm is quite short and is in accordance with a typical Mn=Mn double bond.

Complex **4** crystallizes in the monoclinic space group $P2_1/c$. Figure 2 shows an ORTEP projection[12] of the molecular structure of **4**.

Formally, the 5-hydro-1,4-dimethylnaphthalene ligand occupies one position in the coordination polyhedron. The

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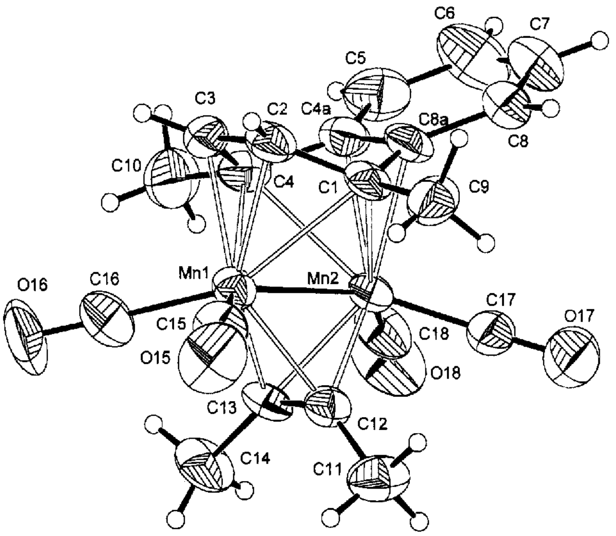
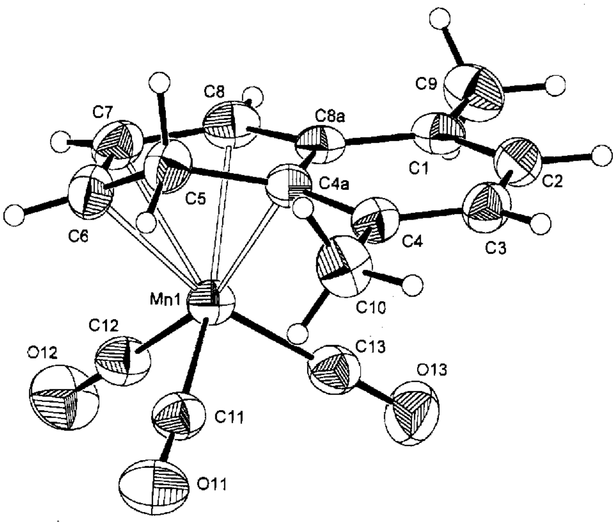
[◇] Part 18: Ref.[1].

Figure 1. Projection of **3**, displacement ellipsoids at the 40-% level[a]



[a]Selected bond lengths [Å]: Mn1−Mn2 2.4132(10), Mn1−C1 2.346(4), Mn1−C2 2.095(4), Mn1−C3 2.113(4), Mn1−C4 2.339(5), Mn1−C12 2.073(5), Mn1−C13 2.051(4), Mn2−C1 2.380(4), Mn2−C4 2.376(5), Mn2−C4a 2.158(5), Mn2−C8a 2.154(4), Mn2−C12 2.009(5), Mn2−C13 2.038(4), C1−C2 1.453(7), C1−C8a 1.441(6), C1−C9 1.498(6), C2−C3 1.413(6), C3−C4 1.420(6), C4−C4a 1.469(6), C4−C10 1.535(7), C4a−C5 1.386(7), C4a−C8a 1.420(7), C5−C6 1.414(9), C6−C7 1.319(9), C7−C8 1.385(8), C8−C8a 1.442(7), C11−C12 1.496(6), C12−C13 1.256(6), C13−C14 1.546(5).

Figure 2. Projection of **4**, displacement ellipsoids at the 40-% level[a]



[a]Selected bond lengths [Å] and angles [°]: Mn1−M1 1.738(6), Mn1−C11 1.798(2), Mn1−C12 1.779(2), Mn1−C13 1.808(3), Mn1−C6 2.189(2), Mn1−C7 2.125(2), Mn1−C8 2.118(2), Mn1−C8a 2.270(2), Mn1−C4a 2.403(2), C1−C2 1.359(3), C1−C8a 1.434(3), C2−C3 1.409(3), C3−C4 1.363(3), C4−C4a 1.424(3), C4a−C5 1.512(3), C4a−C8a 1.435(3), C5−C6 1.505(3), C6−C7 1.397(3), C7−C8 1.402(3), C8−C8a 1.446(3); C11−Mn1−C12 93.60(11), C11−Mn1−C13 96.97(11), C12−Mn1−C13 90.03(11), M1−Mn1−C11 119.8(6), M1−Mn1−C12 125.1(8), M1−Mn1−C13 123.2(3).

three carbonyl ligands and the centre of gravity of the plane formed by C6, C7, C8, C8a and C4a [M1] are arranged as a distorted tetrahedron around Mn1. The distance between Mn1 and the plane formed by the coordinated carbon atoms is 173.9 pm. The coordinated ring 2 is folded along the C4a−C6 axis with a dihedral angle of 142.5°. When the corresponding bond lengths of **4** are compared with those of tricarbonyl(η^5-cyclohexadienyl)manganese[13], the distances between the manganese centres and the carbon atoms common to both rings (C4a, C8a) are longer than those in the parent complex by 18 pm and 12 pm, respectively. This is analogous to the bonding situations in (η^6-benzene)tricarbonylchromium[14] and tricarbonyl(η^6-naphthalene)chromium[15]. In this case, the elongation of the corresponding chromium−carbon bonds is approximately 9 pm in tricarbonyl(η^6-naphthalene)chromium. The alternating bond lengths in ring 1 of **4** and in tricarbonyl(η^6-naphthalene)chromium are almost identical and reflect a slight tendency towards a cyclohexatriene system.

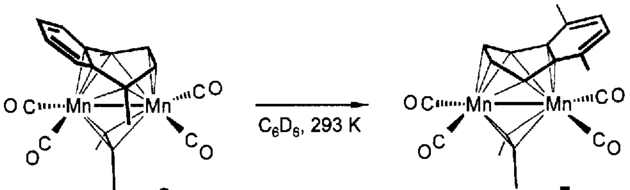
In addition, ^{1}H-NMR spectroscopy of complex **3** shows that both metal centers are coordinated to the methyl-substituted ring 1 of the 1,4-dimethylnaphthalene ligand. The ^{1}H-NMR spectrum of **3** shows three signals between δ = 4.7 and 7.5, each with a relative intensity of two, along with two other signals at higher fields, each with a relative intensity of six.

The singlet with an intensity of six at δ = 0.52 is due to the methyl groups of the 1,4-dimethylnaphthalene ligand, whereas the methyl groups of the bridging alkyne give rise to a singlet at δ = 3.09. The singlet at δ = 4.85 is due to the coordinated 2,3-methine groups. The proton signals of ring 2 form a typical AA′BB′ pattern at δ = 7.32 and 7.40.

On recording the spectrum of **3** at room temperature in C_6D_6, new peaks appeared which have been assigned to tetracarbonyl(μ-$\eta^{2:2}$-2-butyne)(μ-4a,5,8,8a-η:5,6,7,8-η-1,4-dimethylnaphthalene)dimanganese (**5**). In this case, the AA′XX′ proton signals of ring 2 appear at δ = 0.48 for 5-, 8-H and at δ = 4.96 for 6-, 7-H. The signal due to the 1,4-dimethyl groups is now at δ = 1.93 and the singlet due to 2-, 3-H is at δ = 3.17. The chemical shift of the signal due to the methyl groups in the bridging alkyne is only slightly affected by the different coordination modes of the naphthalene ligand.

At room temperature in solution the 1,4-dimethylnaphthalene ligand of **3** shifts, leading to the formation of tetracarbonyl(μ-$\eta^{2:2}$-2-butyne)(μ-4a,5,8,8a-η:5,6,7,8-η-1,4-dimethylnaphthalene)dimanganese (**5**). This rearrangement is probably a result of steric factors. After 14 hours, the conversion of **3** to **5** was complete, with only slight decomposition detected.

Scheme 2



3 **5**

Complex **4** shows nine ^{1}H-NMR signals with relative intensities of 1:1:1:1:1:1:3:1:3. The assignment of the signals to the corresponding protons of the 5-hydro-1,4-dimethylnaphthalene ligand is based on homo-decoupling experiments. The two signals at lowest fields are only slightly shifted with respect to one another. They are due to 2-H and 3-H, and are coupled to each other (J = 7.1 Hz). The signals at δ = 3.46 (*endo*-5-H) and δ = 2.36 (*exo*-5-H) are assigned to the methylene group.

The number of signals observed in the ^{13}C-NMR spectrum was as expected. Their assignment is based on the chemical shifts, the multiplicity and the C–H coupling constants.

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Experimental Section

All operations and reactions were performed under dry, oxygen-free nitrogen. The solvents were dried by standard procedures (*n*-pentane with P_4O_{10} and toluene with sodium), redistilled and stored under nitrogen. Silica gel for column chromatography was heated under vacuum at 413 K, saturated with nitrogen and deactivated with 4 % of water. Photolysis reactions were carried out using a high-pressure mercury lamp TQ 150 (Heraeus-Noblelight, Kleinostheim) operating at 150 W, which was immersed into a 120-ml Duran® vessel with a cooling jacket and wrapped with aluminium foil. During the irradiation, nitrogen was bubbled through the solution in order to mix it. Both the vessel and the column were cooled by a cryostat SK 80 D (Lauda), with methanol as the cooling liquid. – IR: Perkin-Elmer FT-IR 16 PC. – NMR: Bruker AMX 400 (400.13 MHz and 100.62 MHz, for ^{1}H and ^{13}C, respectively). Chemical shifts are given relative to TMS, calculated from the solvent as internal standard ($CHDCl_2$: δ_H = 5.38 and CD_2Cl_2: δ_C = 53.7 ; C_6HD_5: δ_H = 7.20 and C_6D_6: δ_C = 128). – C, H elemental analyses: Perkin-Elmer microanalyser 240. – MS: Finnigan MAT 90. – Pentacarbonyl(μ-$\eta^{4:6}$-1,4-dimethylnaphthalene)dimanganese(*Mn–Mn*) (**1**) was prepared from $[(C_{12}H_{12})Mn(CO)_3][BF_4]$[16] according to a literature procedure[11]. 2-Butyne (**2**) (Fluka) was used without further purification.

Photoreaction of Pentacarbonyl(μ-$\eta^{4:6}$-1,4-dimethylnaphthalene)-dimanganese(Mn–Mn) (**1**) *with 2-Butyne* (**2**): 130 mg (0.32 mmol) of $[Mn_2(\eta^{4:6}-C_{12}H_{12})(CO)_5]$ (**1**) and 0.5 ml (9.2 mmol) of 2-butyne (**2**) were dissolved in 120 ml of toluene in a photoreactor. The dark brown solution was cooled to 193 K and irradiated with UV light. After 45 min, the color of the solution had turned dark red and the carbon monoxide bands of **1** had disappeared. The irradiation was stopped and the solution was concentrated to 2 ml at 4 × 10^{-2} mbar. 4 ml of *n*-pentane was added and the almost black residue was separated into three fractions by column chromatography on silica gel at 233 K. A bright yellow fraction was obtained using *n*-pentane/toluene (5:1) as eluent, from which 10 mg (0.05 mmol) of **4** (16 %) was isolated as a yellow-orange solid. – IR (*n*-pentane; νCO): 2014 (s), 1940 (s), 1929 (s) cm^{-1}. – ^{1}H NMR (CD_2Cl_2, 293 K): δ = 6.90 (dd, J = 7.1, 0.7 Hz, 1 H, 2- or 3-H), 6.89 (dd, J = 7.1, 0.7 Hz, 1 H, 3- or 2-H), 6.44 (dd, J = 5.7, 1.4 Hz, 1 H, 8-H), 5.25 (dddd, J = 7.4, 5.7, 1.1, 1.0 Hz, 1 H, 7-H), 3.46 (ddd, J = 5.7, 1.1, 14.4 Hz, 1 H, *endo*-5-H), 3.26 (dddd, J = 7.4, 5.7, 1.4, 1.3 Hz, 1 H, 6-H), 2.43 (s, 3 H, 1-CH_3), 2.36 (ddd, J = 1.3, 1.0, 14.4 Hz, 1 H, *exo*-5-H), 2.33 (s, 3 H, 4-CH_3). – ^{13}C NMR (CD_2Cl_2, 293 K): δ = 138.39 (s, C-1), 132.71 (s, C-4), 128.73 (d, J = 158.3 Hz, C-2 or -3), 127.78 (d, J = 158.3 Hz, C-3 or -2), 109.08 (s, C-8a), 96.31 (d, J = 165.9 Hz, C-8), 88.68 (s, C-4a), 71.53 (d, J = 173.6 Hz, C-7), 47.43 (d, J = 169.8 Hz, C-6), 25.83 (t, J = 132.1 Hz, C-5), 19.66 (q, J = 126.5 Hz, C-9 or C-10), 18.64 (q, J = 128.8 Hz, C-10 or C-9). – $C_{15}H_{13}MnO_3$ (296.20): calcd. C 60.82, H 4.42; found C 60.7, H 4.6.

The second fraction, which was dark red in color, was eluted with *n*-pentane/toluene (1:1). Removal of the solvent yielded 42 mg (0.10 mmol) of **3** as an almost black solid (62 %). Crystallization from *n*-hexane at 243 K gave dark red crystals which were suitable for crystallography. – **IR** (*n*-pentane; νCO): 1976 (m), 1946 (s), 1904 (s), 1872 (w) cm^{-1}. – ^{1}H NMR (C_6D_6, 293 K): δ = 7.40 (m, J = 6.0, 7.5 Hz, 2 H, 5-, 8-H or 6-, 7-H), 7.32 (m, J = 6.0, 7.5 Hz, 2 H, 6-, 7-H or 5-, 8-H), 4.85 (s, 2 H, 2-, 3-H), 3.09 (s, 6 H, butyne-CH_3), 0.52 (s, 6 H, naphthalene-CH_3). – EI MS (70 eV); *m/z* (%): 432 (1) [M$^+$], 404 (13) [M$^+$ – CO], 376 (33) [M$^+$ – 2 CO], 322 (25) [M$^+$ – 2 CO – C_4H_6], 294 (4) [M$^+$ – 3 CO – C_4H_6], 265 (81) [M$^+$ – 4 CO – Mn], 211 (100) [M$^+$ – 4 CO – Mn – C_4H_6], 156 (34) [1,4-dimethylnaphthalene$^+$]. – $C_{20}H_{18}Mn_2O_4$ (432.24): calcd. C 55.58, H 4.20; found C 54.7, H 4.4.

A third fraction (brown) contained traces of the starting material **1**, which was identified by IR spectroscopy.

Ring-Shift Reaction of **3** *To Give* **5**: 10 mg of **3** was dissolved in C_6D_6 in order to perform an NMR measurement. Within 14 h at room temperature the complex was converted into **5**, with only slight decomposition. – IR (*n*-pentane; νCO): 1976 (m), 1946 (s), 1904 (s), 1872 (w) cm^{-1}. – ^{1}H NMR (C_6D_6, 293 K): δ = 6.99 (s, 2 H, 2-, 3-H), 4.96 (m, J = 5.2, 5.8 Hz, 2 H, 6-, 7-H), 3.17 (s, 6 H, butyne-CH_3), 1.93 (s, 6 H, naphthalene-CH_3), 0.48 (m, J = 5.2, 5.8 Hz, 2 H, 5-, 8-H). – ^{13}C NMR (C_6D_6, 293 K): δ = 143.0 (s, C-1, -4), 130.0 (s, C-12, -13), 128.5 (d, C-2, -3), 95.5 (s, C-4a, -8a), 78.9 (d, J = 177 Hz, C-6, -7), 31.7 (d, J = 163 Hz, C-5, -8), 24.7 (q, J = 130 Hz, butyne-CH_3), 18.8 (q, J = 127 Hz, naphthalene-CH_3). – EI MS (70 eV); *m/z* (%): 432 (2) [M$^+$], 404 (17) [M$^+$ – CO], 376 (34) [M$^+$ – 2 CO], 322 (26) [M$^+$ – 2 CO – C_4H_6], 294 (5) [M$^+$ – 3 CO – C_4H_6], 265 (83) [M$^+$– 4 CO – Mn], 211 (100) [M$^+$ – 4 CO – Mn – C_4H_6], 156 (20) [1,4-dimethylnaphthalene$^+$].

X-ray Structural Analysis: **3**: $C_{20}H_{18}Mn_2O_4$, M = 432.2, orthorhombic space group *Pbca* (No. 61), a = 8.746(1), b = 14.628, c = 29.604 Å, V = 3787.4(8) Å^3 (using 2000 quasi-centered reflections from the whole data set), Z = 8, $D_{calcd.}$ = 1.516 g cm^{-3}, μ = 1.35 mm^{-1}. Crystal size 0.6 × 0.5 × 0.15 mm, T = 293 K, 190 images with a crystal-to-detector distance of 80 mm were collected in the ϕ-range of 0–190 °, 2.71 ° $\le \theta \le$ 23.99 °, 10091 reflections collected, 2825 symmetry-independent reflections (R_{int} = 0.059), 2817 reflections with $I > 0$ used for refinement, 2270 reflections with $I > 2\sigma(I)$. Stoe IPDS diffractometer, graphite monochromator, Mo-K_α (λ = 0.71073 Å), Stoe-IPDS software package (Ver 2.63)[17] for data collection and data reduction. SHELXS-86[18] for structure solution by direct methods and SHELXL-93[19] for refinement of F^2 values by full-matrix least squares. Anisotropic temperature factors were introduced for all non-hydrogen atoms; all hydrogen atoms could be located from difference Fourier synthesis and included in the last stages of refinement using a riding model. 269 parameters were refined, $w^{-1} = \sigma^2(I)$, wR^2 (all) = 0.0668, wR^2 ($I > 0$) = 0.0655, R^1 [$I > 2\sigma(I)$] = 0.0543. – **4**: $C_{15}H_{13}MnO_3$, M = 296.2, monoclinic space group *P*2$_1$/*c* (No.14), a = 7.330(1), b = 6.903(1), c = 26.325(5) Å, β = 93.06(1)°, V = 1330.1(4) Å^3 (38 centred reflections with 13.72° < 2θ < 24.98°), Z = 4, $D_{calcd.}$ = 1.479 g cm^{-3}, μ = 0.99 mm^{-1}. Crystal size 1.0 × 0.5 × 0.2 mm^3, T = 293 K, ω-scan technique, 2.78° $\le \theta \le$ 25.05° ($-1 \le h \le 9$, $-1 \le k \le 8$, $-34 \le l \le 34$), 3589 reflections collected, 2337 symmetry-independent reflections (R_{int} = 0.019), 2208 reflections with $I >$ 0 used for refinement, 1922 reflections with $I > 2\sigma(I)$. Analytical absorption correction using indexed crystal faces was applied to intensity data, min./max. transmission 0.604/0.827; largest difference peak 0.27 e Å^{-3}. Siemens P4 diffractometer, graphite monochromator, Mo-K_α (λ = 0.71073 Å), XSCANS 2.10 b[20] for data collection and data reduction, SHELXS-86[18] for structure solution by direct methods and SHELXL-93[19] for refinement of F^2 values by full-matrix least squares. Anisotropic temperature factors were introduced for all non-hydrogen atoms; all hydrogen atoms could be located from difference Fourier synthesis. The refinement of their positional parameters and individual U values converged with only slow damping. 209 parameters were refined, $w^{-1} = \sigma^2(I)$, wR^2 (all) = 0.0547, wR^2 ($I > 0$) = 0.0525, $R^1[I > 2\sigma(I)] = 0.0299$. – Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-100450. Copies of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [fax: int. code + (1223) 336-033, e-mail: deposit@chemcrys.cam.ac.uk].

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